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Determination of PCB in Dust, Ash and Combustion Gas from City Waste Incinerators

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DETERMINATION OF PCB IN DUST, ASH AND COMBUSTION GAS FROM CITY WASTE INCINERATORS

Ryota Suzuki, Masayuki Ito, Masanori Noma, Akira Moritani, Yuji Watanabe, Tadakatsu Nakaya and Naoki Saito.

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2.2 The Method of Measurement

2.2.1 The wet method. As shown in Figure 2, we first remove water in the exhaust gas in the drain traps whose temperatures are kept to be 0 ∿ 2°C and -20 ∿ -21°C. Then, PCB is trapped in 100 ml of n-hexane which is cooled to 2 ∿ 0°C in parallel in order to prevent the error in the wet gas meter caused by evaporation of n-hexane. Evaporation of n-hexane partly depends on the cooling temperature, but also strongly depends on the suction speed of the exhaust gas. In order for PCB to have sufficient contact with n-hexane, the suction speed was kept below 1 ℓ/min. In this method, the usual problem of icing was made negligible, unless the cooling temperature for removal of water was raised, by adjusting the by-pass cock of the suction pump in the range of the amount of suction up to 300 ℓ. We determined the amount of PCB by adding the amount absorbed in h-hexane, and that in the drain traps and the amount adsorbed to the glass made suction pipe.

2.2.2 The dry method. Figure 3 shows the method used by Tachikawa et al. [5]. The efficiency of collecting PCB considerably depends on the water (about 10 ∿ 35%) contained in the exhaust gas. Therefore, we placed an ice cooled drain trap in front of the florigil column. By this device, water in the exhaust gas was reduced from 30% to 0.8%, according to the measurement illustrated in Figure 3, and its effect on the efficiency of PCB trapping was reduced to a negligible extent as is seen from Table 1.

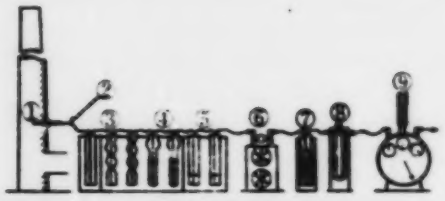

Figure 2. Wet method for the determination of PCB in combustion gas. 1- Glass pipe; 2- Teflon tube; 3- Drain trap 4- PCB impinger; 5- Hexane trap; 6- Suction pump; 7- Oil trap; 8- Olifise* meter; 8- Gas meter.

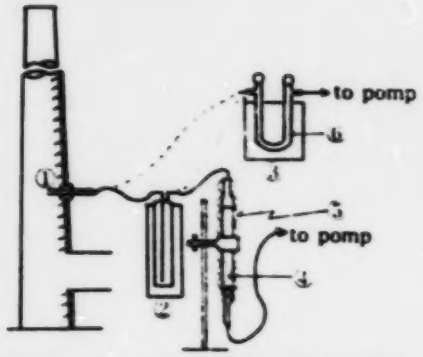

Figure 3. Dry method for the determination of PCB in combustion gas. 1- Glass pipe- 2- Drain trap; 3- U-tube; 4- 5% glycerine-coated florigil column; 5- Aluminum foil; 6- $CaCl_2$.

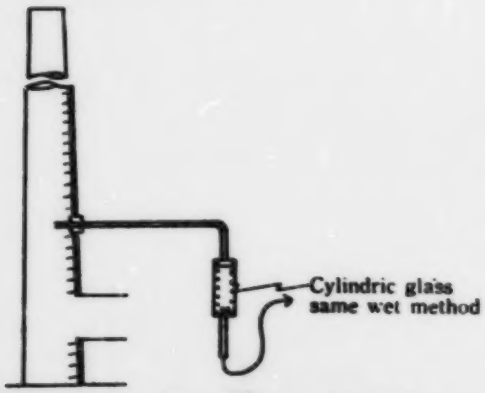

Figure 4. Two shape cylindric glass filter method for the determination of PCB in combustion gas.

Table 1 Effection of Water to determinate PCB Contents

Sample No.	Water (%)	PCB Recovery Rate (%)			Av.
		1	2	3	
1	0	91.1	91.0	93.3	91.1
2	1.0	91.5	92.2	90.3	91.3
3	5.0	88.6	91.3	89.7	89.9
4	10.0	77.8	81.2	72.6	77.2

First, 5 g of florigil for 5% glycerine coating was used. However, florigil even including that near the cock part was polluted to ash color by dust in the exhaust gas. Therefore, we filled up a glass column of 2 cm diameter and 30 cm high with 15 g of florigil. The amount of PCB was determined by adding the amount adsorbed to the column and that in the drain trap and that adsorbed to the glass made suction pipe.

2.2.3 The two shape cylindrical filter method. Figure 4 shows one of the methods of dust measurement adopted by JIS Z8808. The dust collection is done outside the exhaust pipe. In our measurement, in contrast to the measurement of dusts, heating and temperature maintenance of the holder part and the collection pipes are not necessary. Therefore, loss of PCB during its passage through the filter is probably negligible. The amount of PCB is determined by both the amount trapped in the cylindrical filter, and that adsorbed to the holder. We used filtering paper No. 86, with diameter 35 mm and height 12 cm, made by Toyo Roshi.

*Translator's note. This probably pertains to a drying oil meter.

2.3 Apparatus and Reagents.

2.3.1. Reagents

(1) n-hexane: used for testing remaining PCB and agricultural drugs.

(2) Floridin: Made by Floridin Co. 60 - 100 meshes.

(3) Silicagel: Wako Gel S - 1. Made by Wako Drug Co., active at the temperature of 130°C for more than 5 hours.

(4) Standard samples of PCB. KC - 300, 400, 500, 600 made by Wako Drug Co.

2.3.2 Apparatus

Apparatus: Gas chromatograph of type GCSA made by Shimazu.

Comumn: OV-1, 3 mmϕ X 2 m, 2.0% soaked by chromosolve W., 60 - 80 meshes, column temperature 200°C.

DEGS - H_3PO_4 2 - 0.5%. 80 - 100 meshes, column temperature 182°C.

Detector: ^{63}Ni ECD, temperature 250°C.

Carrier gas: Highly purified nitrogen gas 40 ml/min. In addition, we used a circulating current cooling system, Kuderna-Darnish type condensor, and Soksley extractor.

2.4 Method of Analysis

2.4.1 Analysis of PCB contained in burnt ashes, water washed ash, and dusts trapped by the dust collector was done by the method specified by the Environmental Protection Agency [6] for analysis of PCB in soil and the ground. Namely, we added 100 ml of IN-KOH ethanol solution to 20 - 30 g of the sample material and heated and refluxed it for 2 hours. Then, it was extracted by n-hexane and condensed to about 3 ml. It was purified by a silicagel column. After condensing, it was sent for the chromatography. The analysis of PCB in the exhaust gas was done following the specification of the measurement of gas phase PCB given by the Environmental Protection Agency.

2.4.2 We used the decachlorinated method following the PCB analysis method specified by the Science and Technology Department [7]. After reaction, the cleaned and condensed liquid looked yellowish and showed complex peaks before the peak of the decachlorinated biphenyl. This caused errors in quantitative analysis. Therefore, we cleaned the sample up by a florigil column. The net amount of PCB was determined by the following equation

$$A\ (\mu g) = D \times \frac{154.2}{498.7} \times \frac{100}{50} = 0.62 \times D$$

A: Net amount of PCB (μg)

D: Decachlorinated Biphenyl

154.2: Molecular weight of biphenyl

498.7: Molecular weight of decachlorinated biphenyl

50: Assume that the amount of chlorine contained in PCB in the sample to be 50% (Therefore, the amount of biphenyl is 50%).

3. RESULTS AND DISCUSSIONS

The results of analysis in four factories are shown in Table 2.

In the case of the incinerator A, the operation time per day is about 10 hours and the amount of processed wastes is 86 ton/day. The temperature of the exhaust gas is 300°C, water 30%. (This is the value measured in the exhaust pipe after the system of the electric dust collector and monocron.) The amount of the exhaust gas: 44,000 Nm^3/hr, ashes 10 ton/day, the dust collected by the dust collector 90 kg/day, which is equivalent to PCB contents of 0.74 g/day. This value is much lower than the value observed in the case of uncontrolled wastes (leak and evaporation) from the factories using PCG. The amount of PCB in the exhaust gas is also much less than the temporarily determined, acceptable limit of PCB of 0.15 mg/m^3. The amount of dusts in the dust collector is comparatively small. However, the dust collected by the electric dust collector contains 0.2 ~ 0.7 μg/g of PCB. This PCB seems to have been absorbed in dusts and collected by the electric dust collector. We measured PCG contained in dusts in the electric dust collector used in the places other than the incinerator. As shown in Figure 5, we could not detect any PCB in the dust in a cement factory which was collected by the

TABLE 2. PCB CONTENTS FROM INCINERATOR

Incinerator No.	Refuse (ton)	Combustion time (hr)	Combustion gas volume (Nm^3/hr)	PCB Contents				PCB contents/Day
				burned refuse ($\mu g/g$)	multicron ($\mu g/g$)	cottlel ($\mu g/g$)	combustion gas ($\mu g/Nm^3$)	(g)
A	86	10	44,000	0.03	—	0.50	0.88	0.74
B	30	10	44,000	0.02	0.11	0.30	1.35	0.65
C	30	9	31,000	0.08	N.D	N.D	0.32	0.11
D	100	15	92,000	0.15	N.D	N.D	0.82	2.75

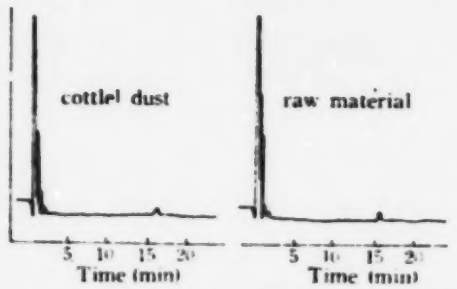

Figure 5. Gas chromatogram of PCB in cement factory. Column: DEGS-H_3PO_4.

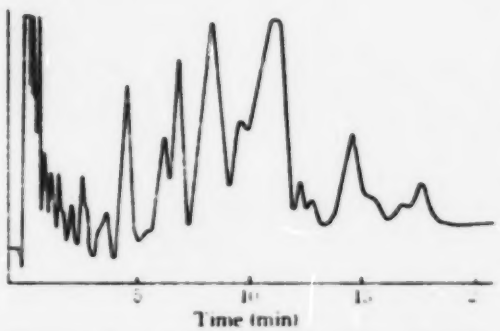

Figure 6. Gas chromatogram of PCB in cottlel dust of certain factory. Column: DEGS-H_3PO_4.

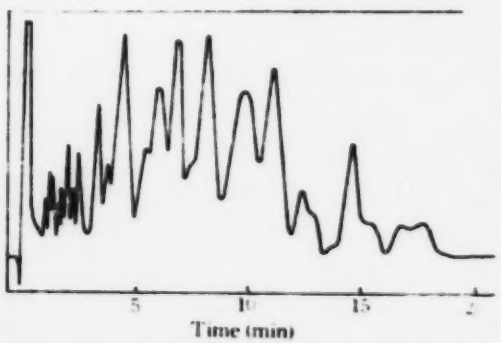

Figure 7. Gas chromatogram of Standart PCB (0.5 ppm) KC300: 400: 500: 600 = 1:1:1:1. Column: DEGS-H_3PO_4.

dust collector and also in the raw material. However, as is seen in Figure 6, dust from a certain factory showed a peak pattern which agreed well with the pattern in Figure 7, and the dust contained 0.70 ∿ 1.1 μg/g of PCB.

In the case of decachlorinated samples, we used gas-chromatographs of column OV-1 and DEGS H_3PO_4 and compared them with the standard sample. As is shown in Figure 8, Rt was in good agreement. The PCB concentrations determined by the peak height method and the decachlorinated method were compared with each other.

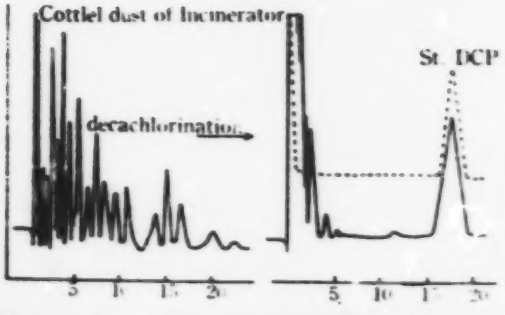

Figure 8. Gas chromatogram of decachlorinated PCB in cottlel dust of incinerator. Column: OV-1.

The amount of PCB in the exhaust gas determined by the decachlorinated method was about 70 - 80% of that measured by the peak height method. However, in the case of dust collected by the dust collector, the decachlorinated method showed values 3 ∿ 10 times higher than those determined by the peak height method. This is probably due to the effect of biphenyl contained in the sample [8]. Therefore, further investigation is necessary for the use of decachlorinated method in the samples from incinerators.

Table 3 Cl Number Distribution in Combustion gas and Dust

Sample and Method	Cl_2	Cl_3	Cl_4	Cl_5	Cl_6	Cl_7	Cl_8	PCB(μg Nm³)
Comb. gas Wet Method	4.5	5[illegible]	18.6	6.0	8.7	6.8	4.[illegible]	[illegible].54
"	2.8	4[illegible]	29.6	9.9	5.4	1.5	1.[illegible]	12.[illegible]
Dry Methnd	2.4	56.2	23.7	4.7	8.2	2.2	1.[illegible]	3.25
"	0.9	20.1	40.1	15.0	8.1	9.8	[illegible]	1.70
Two shape filter Method	4.6	56.2	17.2	14.0	3.7	4.1	—	0.35
"	1.1	48.1	36.0	4.1	4.9	2.2	2.[illegible]	3.60
Powder Dust Low Volume Air Sampler	0.5	35.4	27.1	8.8	13.3	11.7	3.1	0.0025
Burned Refuse	1.5	2[illegible]	25.3	11.2	11.0	11.0	11.4	[illegible].010(μg g)
Cottlel Dust *	3.2	12.5	28.2	16.5	25.1	11.4	3.1	0.70
" **	1.7	2[illegible].7	16.2	10.6	14.0	15.8	12.2	0.56

* Cottlel Dust in Incinerator

** Cottlel Dust in a certain Factry

Table 4 Variation of PCB Contents on Time passes

Sampling Date	Cottlel Dust (μg g)	Burned Refuse (μg g)	Comb gas (μg Nm³)
47. 10. 19	[illegible]	0.11	—
48. 2. 28	—	—	[illegible]
48. 5. 15	0.5	0.03	[illegible]
48. 8. 23	[illegible]	0.04	—
48. 9. 29	[illegible]	0.04	—
48. 11. 27	0.5[illegible]	0.01	—

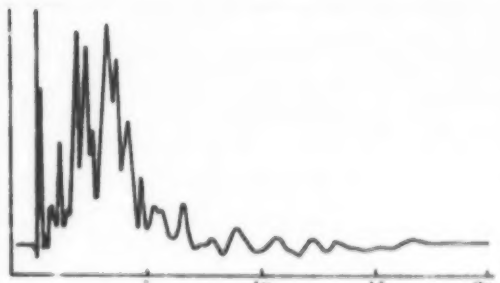

Figure 9. Gas chromatogram of PCB in combustion gas with two shape cylindric glass filter method.

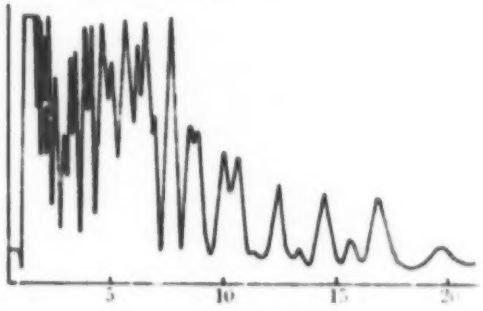

Figure 10. Gas chromatogram of PCB in combustion gas with wet method. Column: OV-1.

Table III shows the results computed from the data of the ratio of PCB composites for those from KC-200 up to KC-800, which were measured by Kashimoto [9] et al., using the method of numerical evaluation of PCB. If we divide the chlorine number into those between 2 ∿ 5 and those between 6 ∿ 8, Figures 9 and 10 show that PCB of low chlorine numbers dominates in the exhaust gas. In the dusts from the dust collector, the chlorine number of PCB is relatively uniformly distributed. The chlorine number of PCB contained in the fine dusts trapped by the low volume air sampler shows the distribution between these of the exhaust gas and of dusts from the dust collector.

Table IV shows the results of study of time variation of the discharged amount of PCB. Since the nature of wastes varies constantly, differing from the case of the crude oil boiler and the calcination furnace which always use a certain amount of fuel, the combustion temperature of the furnace and its load vary significantly. Therefore, accurate measurements are rather difficult. However, PCB in the dust collected in the dust collector temporarily decreased and again increased. At present, the direction of its change is uncertain.

Table 5 Compared PCB Contents with Different Sampling Method

Sampling Method	PCB Contents (μg Nm³)					
	1	2	3	4	5	6
Wet Method	0.83,1.31,1.80,2.12	2.35,1.45,2.03	1.07,1.08	0.97	0.41	12.06
Dry Method	1.73,2.20	2.09	0.64,0.73	0.87,0.91	0.72,0.30	3.25
Two shape filter Method	—	—	—	—	0.35	3.96

We compared the methods of sampling PCB in the exhaust gas, using Table V and considering the circumstances of sampling.

The wet method: Since we use h-hexane, danger of fire is great. Cooling takes time. A part of the sampled PCB may be lost by its adsorption to the wall.

The dry method: Set-up of clean absorption columns is rather difficult. There are some problems of PCB dissolving in alcohol after its sampling.

The two shape cylindrical filter method: Cleaning of the cylindrical filter used in this method and extraction of PCB after sampling can be easily done by using the Socksley extractor. The load is small and the apparatus is rather simple. After sampling, the inner wall of the cylindrical filter is covered with the layer of dust of blackish grey color. We examined the leak of PCB through the filtering paper by connecting the wet method apparatus after the two shape cylindrical filtering paper holder. However, we did not detect any PCB. Therefore, this method seems most suited for sampling PCB in the exhaust gas.

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SUMMARY

Determination of PCB in Dust and Combustion Gas from City Wastes Incinerators.

Ryota SUZUKI, Masayuki ITO, Masanori NOMA, Akira MORITANI, Yuji WATANABE, Tadakatsu NAKAYA and Naoki SAITO

Polychlorobiphenyl (PCB) contents in burned refuse, water washed ash, dust collected with multicron or electrostatic precipitator and combustion gas were estimated.

For the sampling of PCB in the combustion gas, the adsorption into n-hexane by impinger as the wet method, the adsorption by florigil colume coated with 5% glycerin as the dry method and the method of two shape-cylindrical glass filter were investigated. The most effective method was last one.

PCB contents in combution gas were variated from none detect to 12μg/Nm^3, in burned refuse, water washed ash and dust were 0.01-0.70 ppm g.